CHAPTER IV

EXPERIMENTAL

4.1. Materials

Polymeric matrix used in this study is polypropylene grade HP 644T obtained from HMC Polymers Company LTD., Thailand. It has a melt flow index (MFI) of 60 g/10 min and density of 0.9 g/cm^3. A compatibilizer is maleic anhydride grafted polypropylene (PP-g-MA) grade Fusabond P MZ203D from Dupont Company, LTD., Thailand. The PP-g-MA has a melt flow index (MFI) 102 g/10min and density of 0.94 g/cm^3. The filler used is rubber woodflour, having average particle size of 250-300 micrometer. It is a waste material from a saw-mill factory in Thailand.

4.2 Methodology

4.2.1 Preparation of Polymer Matrix and Compatibilizer

The polymer matrix and the compatibilizer for this research are polypropylene (PP) maleic anhydride grafted polypropylene (PP-g-MA), respectively. They were dried in an air-circulated oven at 105°C for 24 hours before use.

4.2.2 Preparation of Woodflour

The woodflour for this research is rubber wood (*Hevea brasilienes*). It was selected for this work because of its availability and this wood is widely grown in Thailand. The wood sawdust was crushed using a ball mill apparatus to reduce its particle size to 250-300 micrometer. The density of the woodflour determined using a gas pycnometer is 1.49 g/cm^3 (Jubsilp et al., 2005). The wood flour was dried in an oven at 105°C for 24 hours before use.

4.2.3 Preparation of Wood Composites

The proportion of polypropylene to woodflour for this research was fixed at 60:40 by weight. Two experiments were performed in this study. In the first experiment, polypropylene was mixed with wood flour in a twin screw extruder (Rheocord 300p of Haake Inc.) with rod capillary die at 170°C using the screw speed of 50 rpm. The compound was obtained in a pellet form. After drying at 105°C for 2 hours, the sample was injection-molded using the maximum temperature of 180°C. The dimension of the bar-shaped specimens is 20x115x3.5 mm^3.

In the second experiment, polypropylene was mixed with the wood flour and PP-g-MA (3% by weight) in a twin screw extruder at 170°C with the screw speed of 50 rpm. The sample was further processed by an injection machine to a standard bar and dogbone shape.

All specimens were irradiated with γ-rays (gamma rays) from a $_{60}Co$ source at the facility of the Office of Atom for Peace, Thailand. Radiation doses of 5, 10, 20 and 30 kGy with a dose rate of 0.2112 kGy/min, at room temperature were evaluated. The "dose" or the amount of radiation energy imparted to the matter was determined based on calorimetric measurements following IAEA standard. Energy absorbed in a calorimeter contained a material having radiation absorption characteristics similar to those of the target material irradiated with the same beam characteristics (energy and current). The specimens were irradiated under two atmospheres i.e. nitrogen (nitrogen purging for 15 min before irradiated), and air atmosphere.

4.3 Wood Composite Characterization

4.3.1 Density

Density determination was performed by Archimedes' principle. This principle states that every solid body immersed in a fluid apparently loses weight by an amount equal to that of the fluid it displaces. The specimens were weighed in air

atmosphere and were immersed and weighed again in water. The density can be calculated from the two weights as follow:

$$\text{Density, } \rho \text{ (g/cm}^3\text{)} = \left[\frac{A}{(A - B)}\right] \times \rho_0 \qquad (4.1)$$

Where:

ρ is density of the solid

A is weight of the solid in air

B is weight of the solid in the liquid

ρ_0 is density of the auxiliary liquid at the given temperature (this value depends on the temperature and must be taken from a density table)

4.3.2 Mechanical Characterization

4.3.2.1 Flexural Property Measurement

Flexural modulus and flexural strength of the composite specimens were measured by a universal testing machine (Instron Instrument, model 5567) according to ASTM D790. Three-point bending test was carried out at room temperature at the crosshead speed of 1.2 mm/min with the support span of 48 mm. The dimension of the specimen was 20×60×3 mm^3. Five specimens from each composite composition were examined and the average values were reported.

4.3.2.2 Tension Property Measurement

Tensile modulus and tensile strength of the specimens were obtained by a universal testing machine (Instron Instrument, model 5567) according to ASTM D638. The test specimens are a dumbbell shape with a uniform thickness. They were tested using a crosshead speed of 5 mm/min with the pre-load of 100 N giving a straight tensile force. The tensile modulus defined as the ratio of stress to strain was

determined from the initial slope of the stress-strain curve whereas the tensile strength is the ultimate stress. Five specimens from each composition were tested with the average values reported.

4.3.2.3 Creep Property Measurement

Creep tests under tension were also performed on a universal testing machine (Instron Instrument, model 5567). Specimens of the same sizes as the tensile test specimens were chosen. Each specimen was stretched with a constant load at 40% of the tensile strength. The time period used for the creep test was up to 6 hr and the tensile strain was recorded as a function of time. The fractional deflection is usually used as a relative measurement of creep performance. It was determined by a following equation:

$$\text{Fractional deflection } (F_d) = \frac{C_i}{C_0} \qquad (4.2)$$

Where:

F_d is fractional deflection during the period i

C_i is the creep deflection during the period i (mm)

C_0 is the instantaneous deflection (mm)

4.3.3 Thermal Characterization

4.3.3.1 Differential Scanning Calorimetry (DSC)

Thermal characteristic of composite specimens was examined using a Differential Scanning Calorimeter (model 2910, TA Instrument). The weight of the sample was 5 mg. Each sample was encapsulated in a non-hermetic aluminum pan. The experiment was performed at the heating rate of 10°C/min from 30 to 210°C under nitrogen purging.

4.3.3.2 Thermogravimetric Analysis (TGA)

Thermal decomposition behaviors of each specimen such as degradation temperature and weight loss of the samples were determined by using Thermal Gravimetric Analyzer, TGA (Model TGA 851), from Mettler Toledo. The experiment was performed under nitrogen purging with a constant flow of 60 ml/min. Sample mass of about 10 mg was heated using a linear heating rate of 20°C/min from room temperature to 800°C.

4.3.4 Interfacial Interaction

The interfacial interaction or the adhesion between wood flour filler and polypropylene polymer matrix was examined using SEM micrographs. The micrographs were obtained using Scanning Electron Microscope (Model JSM-5800LV, Jeol), at an acceleration voltage of 15 kV. The fracture surface of each specimen was coated with thin gold film, of which the thickness was between 10 to 20 nm, prior to obtaining the micrograph.

4.3.5 Extraction Percentages Examination

Extraction percentages of the polymer matrix and wood composites were evaluated using p-xylene extraction according to ASTM D2765 (Method C). The specimens were cut to small pieces of approximately 0.500±0.020 g. They were weighed before immersion in 30±0.1 ml of hot xylene for 24 hr. The extracted specimens were later dried at 150°C and re-weighed until a constant weight was obtained. Percent extraction was determined according to the following equation:

$$\text{Extraction (\%)} = \frac{(W_s - W_d)}{W_o} \times 100 \qquad (4.3)$$

Where:

W_s is weight of specimen being tested

W_d is weight of dried gel

W_o is original polymer weight

(the amount of polymer in the specimen being tested)